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**(NASA-TM-86938) HIGH TEMPERATURE PROPERTIES
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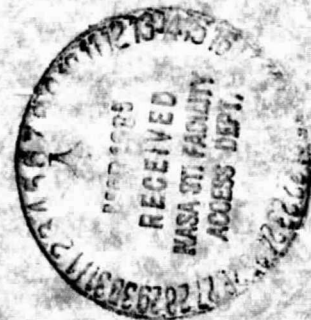
High Temperature Properties of Equiatomic FeAl with Ternary Additions

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NASA

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SUMMARY

The aluminide intermetallic compounds are considered potential structural materials for aerospace applications. The B2 binary aluminide FeAl has a melting point in excess of 1500 K, is of simple cubic structure, exist over a wide range of composition with solubility for third elements and is potentially self-protecting in extreme environments. The B2 FeAl compound has been alloyed with 1 to 5 at % ternary additions of Si, Ti, Zr, Hf, Cr, Ni, Co, Nb, Ta, Mo, W, and Re. The alloys were prepared by blending a third elemental powder with pre-alloyed binary FeAl powder. Consolidation was by hot extrusion at 1250 K.

Annealing studies on the extruded rods showed that the third element addition can be classified into three categories based upon the amount of homogenization and the extent of solid solutioning. Constant strain rate compression tests were performed to determine the flow stress as a function of temperature and composition. The mechanical strength behavior was dependent upon the third element homogenization classification.

INTRODUCTION

The equiatomic B2 aluminides of Fe, Ni, and Co are considered potential component and structural materials for diverse applications in power systems of aircrafts and automobiles, fossil fuel conversion technology, heat exchangers, and recuperators (refs. 1 and 2) where a high modulus, high tensile strength and high temperature creep strength as well as exceptional oxidation resistance of these intermetallic compounds can improve operation efficiency, reduce component weight, and save strategic elements such as Ta, Nb, and Cr. Knowledge of the mechanical behavior of the FeAl, NiAl, and CoAl intermetallics is quite scant and the effects of composition, temperature, and third element additions are virtually unknown. However, it has been demonstrated that FeAl (ref. 3) and NiAl (ref. 4) are ductile at elevated temperature, but not very strong while CoAl (ref. 5) is strong but not very ductile at elevated temperatures. None of the three intermetallics have exhibited commercially acceptable ductility (>5 percent) at room temperature in tension. Since these B2 binary aluminide systems exist over a range of compositions (fig. 1) and do possess solubility for third element additions, they offer the potential of producing an acceptable material by alloying. That is, third element additions to induce room temperature ductility, or conversely, third element

additions to increase elevated temperature strength whichever is more critical for a particular application.

PREVIOUS RESEARCH ON BINARY FeAl

The focus of the research effort of the NASA Lewis Research Center on the ordered alloy FeAl, a B2 aluminide, has been alloying with a third element using pre-alloyed binary powders, consolidation by hot extrusion, and then evaluation of the deformation behavior as a function of composition, grain size and temperature. In an effort to understand and realize improvement in mechanical properties of the ternary FeAl compositions, the P/M binary FeAl has been studied extensively at the Lewis Research Center. Clark and Whittenberger (ref. 6) studied the thermal expansion of the B2 FeAl in air as a function of composition. Whittenberger (refs. 7 and 8) has shown that for the Fe-39.8 Al¹ binary, thermomechanically processed by hot extrusion of pre-alloyed powder, the slow plastic deformation behavior was a strong function of grain size; where small grain size resulted in stronger materials. Constant velocity slow compressive plastic flow properties of FeAl materials ranging in composition from about 42 to 49 at % Al were also studied by Whittenberger (ref. 9) as a function of grain size and temperature. It was shown that two independent deformation mechanisms with the same apparent activation energy exists, both dependent on grain size which exhibits a stronger influence on strength than composition. In summary, Whittenberger has characterized the binary B2 FeAl material produced by hot extrusion of pre-alloyed powders and modeled its compressive slow plastic deformation mechanism.

Alloy Preparation

Strengthening of the near equiatomic FeAl (and NiAl) material with elemental ternary additions to the pre-alloyed binary intermetallic powders has been investigated by Vedula and Titran and co-workers (refs. 10 and 11). A series of ternary FeAl alloys were produced by powder metallurgy techniques. The alloying additions were selected on the basis of past experience in the development of BCC refractory metal alloys and nickel-base superalloys. The selected ternary additions are shown outlined on a Periodic Table in figure 2. These alloying additions were added at the 1 to 5 at % level. It was recognized, based on available ternary phase diagrams, that all of the third element additions would not have equivalent solubilities in FeAl. The blended powders were canned in mild steel and extruded at 1250 K at a reduction ratio of 16:1. The nominal chemical compositions of the extruded FeAlX materials are listed in table I. After hot extrusion, the mild steel cans were leached off in 50:50 nitric acid:water. The 1 and 2 at % ternary alloys were annealed for 100 h at 1425 K. The 5 at % alloy material was heat treated for 175 h at 1525 K in an attempt to maximize the diffusion of the ternary addition into the binary matrix.

¹All compositions are given in atomic percent.

RESULTS

Microstructural Characterization

Microstructural examination of the as extruded and heat treated materials indicated that they could be classified in three rather broad categories based on the extent to which the 5 at % ternary element had homogenized. Class I FeAlX alloys are single phase materials after homogenization and included the elements Ni, Co, Ti, Si, Mn, and Cr. The solubilities of these elements is consistent with the consideration of atomic size (ref. 12) and published phase diagrams. Figure 3 shows a typical single phase structure of FeAl5Ti.

The other elemental 5 at % alloying additions studied showed incomplete solubility even after 175 h of diffusion at 1525 K. In some instances the volume fraction of the second phase appears to have increased during homogenization. This indicates the diffusion of the matrix Fe and/or Al into the elemental powder particle resulted in the formation of either a binary or ternary intermetallic particle in a FeAl matrix with an apparent higher volume fraction of particles than the original extrusion.

The ternary additions which exhibited this type of significant interdiffusion without resulting in a single phase microstructure are classified into a Class II category. Class II elements are Zr, Hf, Nb, Ta, and Re. Figure 4 shows the microstructure of the FeAl5Nb alloy both as extruded and homogenized. It is evident that the volume fraction of second phase particles has increased substantially.

The third category, Class III, consists of those elements which do not exhibit any significant interdiffusion. Mo and W are examples of Class III ternary element additions to FeAl. The original Mo and W powder particles remain essentially as they were for the extruded condition following the homogenization treatment. Figure 5 illustrates this category for the FeAl5 Mo.

The 5 at % ternary alloying additions have been classified into three categories based on the extent of their observed interdiffusion with FeAl as listed in table II. This classification will be shown to have an important bearing on the slow plastic deformation behavior at elevated temperatures.

Deformation Behavior

The elevated temperature (1100-1300 K) slow plastic deformation behavior of FeAl with ternary additions was studied by using constant velocity compression tests on right circular cylinders. The stress-axis was parallel to the extrusion axis.

Right cylindrical specimens 5 mm in diameter ranging in length from 10 to 12 mm were machined from decanned and homogenized materials. The extruded bar stock was cut into specimen slugs and both decanned, homogenized, and then centerless ground to 5.0 mm diameter. Specimens 12.5 mm in length were surface ground to length (10 mm minimum) with both ends flat, parallel and true to center axis of the 5 mm cylinder. In general, grinding produced crack-free surfaces.

Elevated temperature compression tests were conducted in air at 1100, 1200, and 1300 K which range from about 0.7 to 0.8 T_m . Testing was accomplished at constant velocities ranging from 2.12×10^{-2} to 2.12×10^{-5} mm s⁻¹ in a universal testing machine where the load was transmitted to the specimen through SiC push rods. Pieces of tungsten foil 25 μ m thick were placed between the ends of the test specimen and such rods to prevent welding. Temperature was monitored with type R thermocouples wired to the top and bottom of the test specimens. During testing, the temperature was maintained to within 1 K of the set point, and the difference between the two thermocouples never exceeded 3 K. Specimens were slowly heated to temperature over 2+h while a small stress (about 5 MPa) was applied by operating the test machine in the load cycle mode to retract the expanding push bars and to maintain specimen alignment.

Both stress and strain data were calculated from the load-time charts. Strains were determined either by the offset method (ref. 13) when an elastic region could be discerned or by crosshead displacement when little elastic deformation could be seen. In general the calculated strain at the end of a test agreed well (± 10 percent) with that determined by the measured change in length; however, for consistency, all calculated strains were normalized to the actual length change. The true stresses were computed on the basis of conservation of volume and uniform deformation.

The 5 at % ternary addition alloys of FeAl were initially compression tested at 1300 K to assist in the ranking of the deformation strength of these compositions. The results of the 1300 K compression tests are shown in figure 6. The slopes of the lines which are for clarity only were made parallel to existing FeAl data (refs. 7 to 9). It is noted that the Zr alloys, 48Fe47Al5Zr and 56Fe39Al5Zr have a flow resistance greater than a factor of three times that of the binary FeAl at a comparable grain size, 20 μ m. The 49Fe46Al5Nb and 5Mo alloys both appear to have better than a factor of six greater strength than the binary FeAl at a comparable grain size. It is noted that the strongest ternary alloys are those with either a Class II or Class III addition. That is, the volume fraction of second phase appears to have increased due to the apparent diffusion of Fe and/or Al into the elemental particle during the homogenization cycle for a Class II, or no noticeable change in volume fraction of second phase for a Class III. Most of the single phase Class I alloys did not exhibit significant improvements in the slow plastic compressive flow strength when compared to the binary alloys. However Ti and Si increased the strength more than the Class II W addition. Based upon the work of Whittenberger (ref. 9), we are quite confident the strength increases noted are due to the 5 at % ternary addition and not a grain size effect or Fe:Al ratio effect.

Additional test specimens machined from the extruded bar stock were annealed at 1425 K for 100 h which is sufficient to homogenize the Class I compositions. In addition 0.8 boron alloys were prepared by extrusion and annealed at 1325 K for 100 h. The results of constant strain rate compression tests on the 56Fe39Al5Zr as a function of test temperature is shown in figure 7 and is compared to the binary FeAl. At a constant strain rate of 2×10^{-6} s⁻¹, at 1100 K, the 5Zr addition produces a flow stress greater than 120 MPa compared to only 20 MPa for FeAl. The 5Zr composition shows a 200 K temperature advantage over the FeAl binary. The results of constant strain rate compression tests on the nominal 0.8B addition to the 52Fe48Al binary as a function of test temperature is shown in figure 8 along with comparable binary data.

At 1100 K and a strain rate of $2 \times 10^{-6} \text{ s}^{-1}$ the 0.8B alloy has a flow stress of about 80 MPa whereas the FeAl binary has a flow stress of about 20 MPa. The strength improvement of the boron addition is shown to exist to at least 1300 K, but seems to decrease slightly with increasing test temperature. The results of constant strain rate compressive tests at 1100 K on various ternary FeAl-X alloys is shown in figure 9 and reflects a portion of the results of this ongoing screening study.

CONCLUSIONS

The results of constant strain rate compression tests clearly indicate that three of the commonly accepted strengthening mechanisms operate in the B2 FeAl intermetallic system containing ternary additions:

1. Solid solution strengthening - The Class I alloys containing additions of 1 to 5 at % Cr, Ti, Mn, Fe, and Co all exhibit higher flow stress than a comparable FeAl binary at a constant strain rate and a single phase microstructure.

2. Second phase strengthening - The Class II alloys containing additions of 0.8 to 5 at % B, Zr, Ta, Nb, Re, and Hf all exhibited higher flow stress than comparable FeAl tests. It is believed that to some extent, these ternary additions did go into solution in the matrix and reprecipitate possibly as a ternary intermetallic compound which effectively pin dislocations. The Class III ternary additions, Mo and W, which were less effective in strengthening are thought to behave as inert second phase particles which may hinder grain boundary sliding as would the larger particles in the Zr, Ta, Nb, and Hf alloys.

3. Grain boundary strengthening - The 0.8 at % B addition has a remarkable strengthening effect on FeAl. It has been postulated that boron in the Fe_3Al (ref. 14) and Ni_3Al (ref. 15) systems improves the grain boundary cohesion. The extremely high strength noted for the FeAl0.8B alloy may also reflect a synergistic effect of formation of boride precipitates on dislocation lines along with restriction of grain boundary sliding.

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TABLE I. - NOMINAL COMPOSITION
IN ATOMIC PERCENT OF
CONSTANT STRAIN RATE
COMPRESSION TESTED
FeAlX MATERIALS

Specimen	Nominal composition		
	Fe	Al	X
Fe-31 (B)	52	48	0.8
Fe-14 (Si)	56	39	5
Fe-22 (Ti)	56	39	5
	50	48	2
Fe-24 (Cr)	49	46	5
Fe-27 (Co)	49	46	5
Fe-40 (Zr)	56	39	5
	50	48	2
Fe-41 (Nb)	49	46	5
	50	48	2
Fe-42 (Mo)	49	46	5
	49	50	1
Fe-73 (Ta)	49	46	5
	50	48	2
Fe-74 (W)	49	46	5
Fe-75 (Re)	49	46	5
	49	50	1

TABLE II. - CLASSIFICATION OF TERNARY
ALLOY ADDITIONS FOR 5 AT % THIRD
ELEMENT ADDITIONS TO FeAl AFTER
HEAT TREATING FOR 175 h at 1525 K

Class	Third element addition	Solubility
I	Fe, Cr, Mn, Co, Ti	Total
II	Nb, Ta, Zr, Si, Hf, Re	Partial
III	W, Mo	None

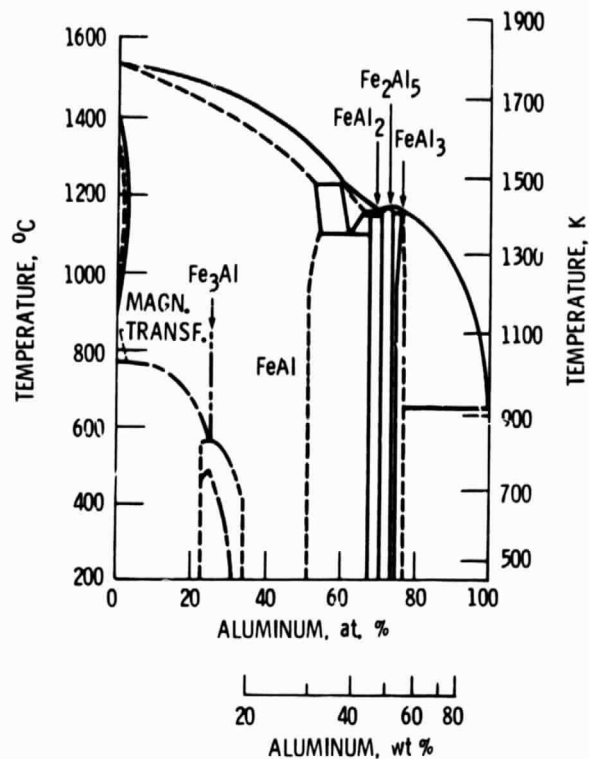


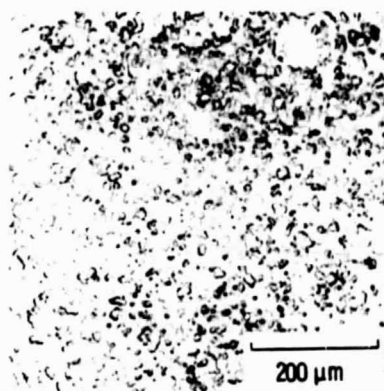
Figure 1. - The iron-aluminum phase diagram showing the large composition range of the B2 phase.

H																	He	
Li	Be											B	C	N	O	F	Ne	
Na	Mg											Al	Si	P	S	Cl	Ar	
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr	
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe	
Cs	Ba		Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn	
Fr	Ra																	
		LANTHANIDE SERIES																
		ACTINIDE SERIES																
		La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu		
		Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr		

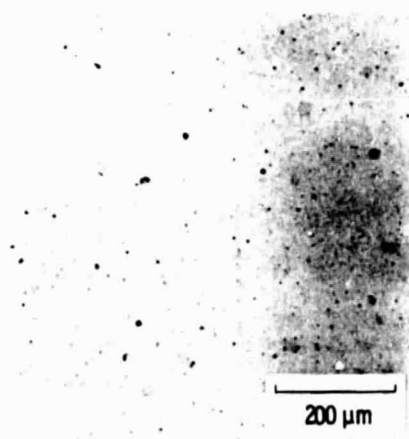
Figure 2. - The periodic table with the elements used for ternary additions in this study outlined.



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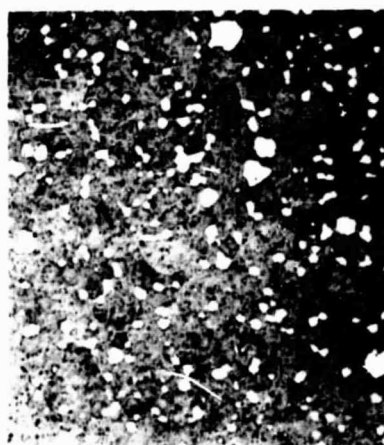


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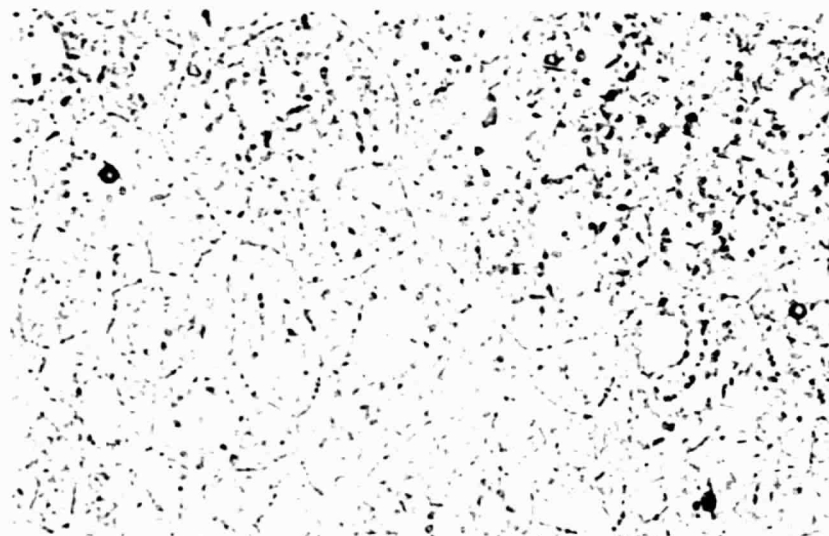
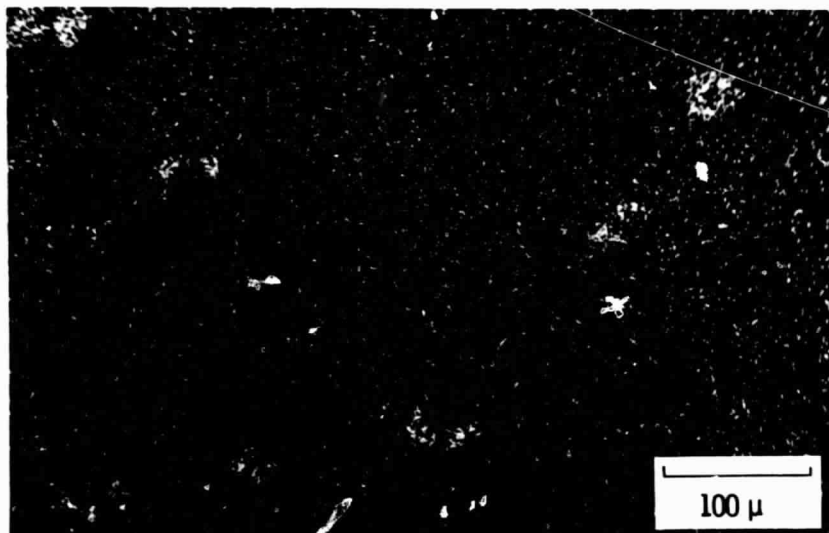
Figure 3. - Photomicrograph showing the solid solutioning of a Class I alloy following the 1425 K - 175 h heat treatment (transverse section).



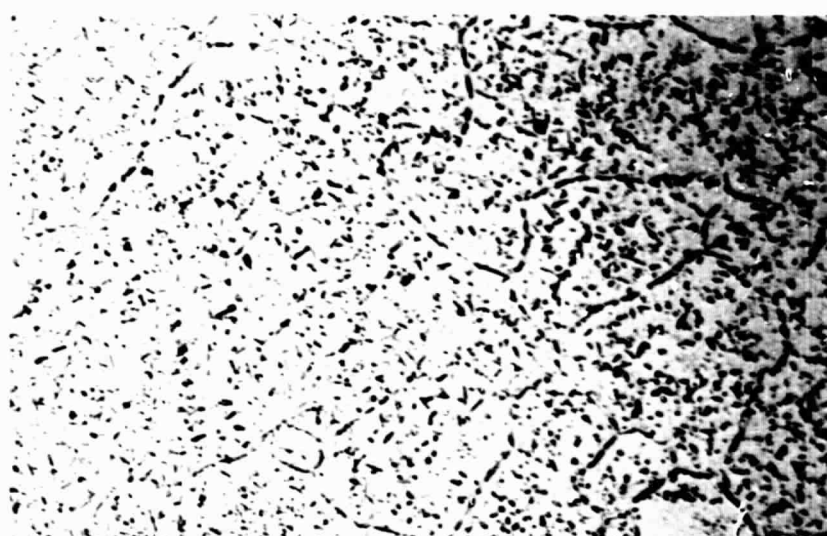
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Figure 4. - Photomicrograph showing the increase in volume fraction of Nb rich phase of Class II alloy following the 1425 K - 175 h heat treatment (transverse section).

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Figure 5. - Photomicrograph and x-ray map showing the relative constant volume fraction & distribution of the Mo particles of a Class III alloy following the 1425 K - 175 h heat treatment (transverse section).

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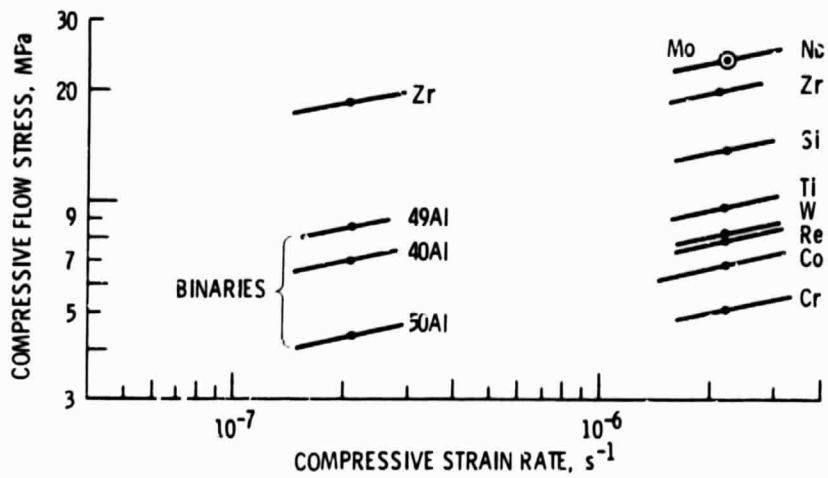


Figure 6. - Comparison of true compressive strain rate against flow stress for various 5 atom percent third element additions to FeAl. Test temperature was 1300 K.

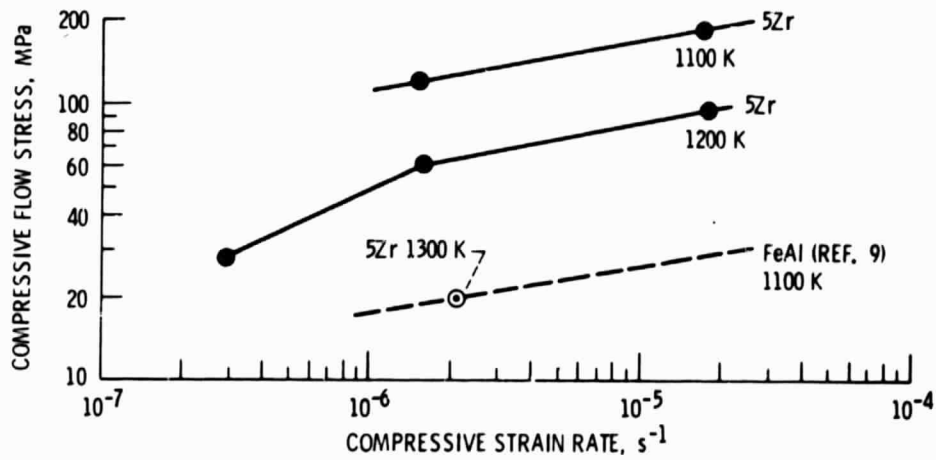


Figure 7. - Compressive flow stress/strain rate behavior for the FeAl-5Zr alloy as a function of temperature.

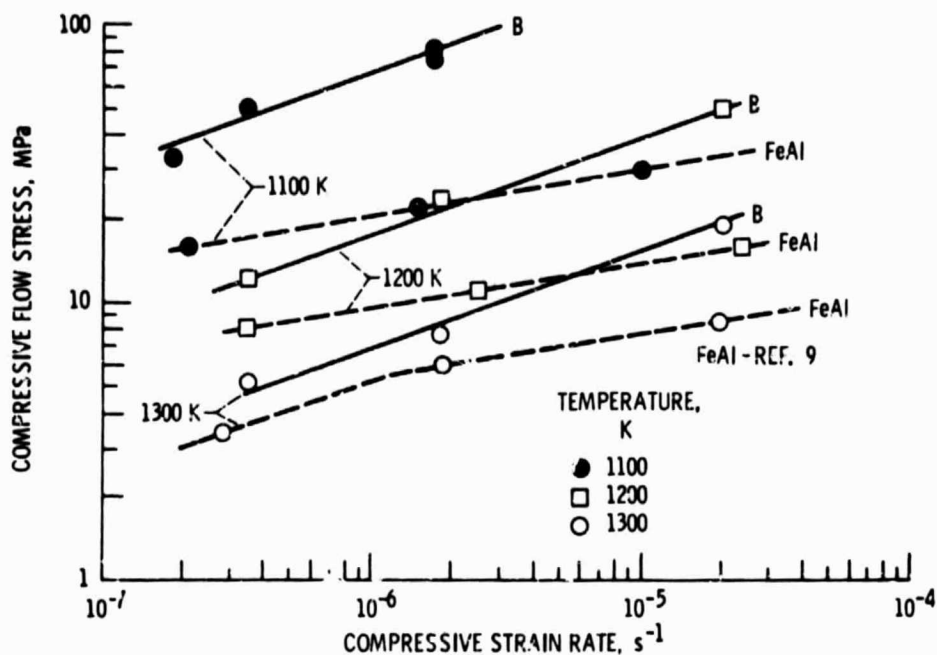


Figure 8. - Compressive flow stress/strain rate behavior of the FeAl-0.8B alloy and comparable FeAl material as a function of temperature.

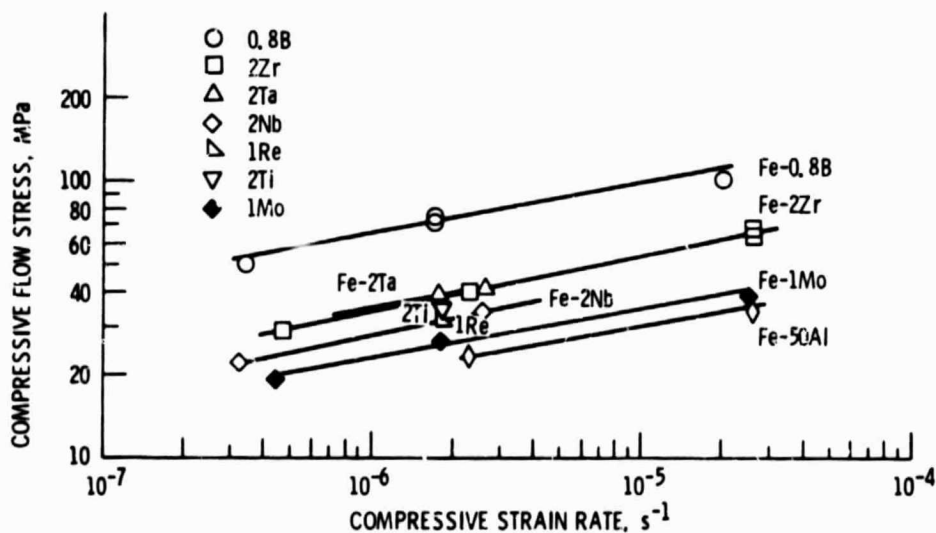


Figure 9. - Comparison of the compressive flow stress/strain rate behavior for several FeAl-X materials at 1100 K. (Numbers before chemical symbols indicate third element concentration in atom percent. Material heat treated for 100 hr at 1425 K prior to testing.)